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IS 12125 (1987): Benzotriazole, photographic grade [CHD 5: Electroplating Chemicals and Photographic Materials]

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SPECIFICATION FOR BENZOTRIAZOLE, PHOTOGRAPHIC GRADE

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MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Gr 4

December 1987

Indian Standard

SPECIFICATION FOR BENZOTRIAZOLE, PHOTOGRAPHIC GRADE

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(*Continued on page* 2)

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Indian Standard

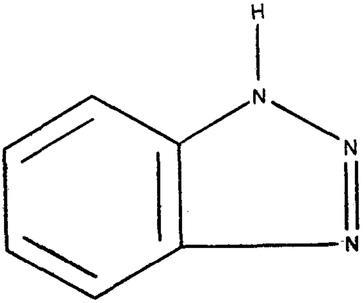
SPECIFICATION FOR BENZOTRIAZOLE, PHOTOGRAPHIC GRADE

0. FOREWORD

0.1 This Indian Standard was adopted by the Bureau of Indian Standards on 30 June 1987, after the draft finalized by the Photographic Materials Sectional Committee had been approved by the Chemical Division Council.

0.2 This Indian Standard is one of a series of specifications for photographic grade chemicals which are commonly used in the processing of sensitized photographic materials. These specifications have been prepared to establish criteria of purity which will provide a practical and economical grade and prevent possible faulty processing which might be caused by chemicals of inferior quality and to furnish manufacturers, suppliers and processors with reliable and readily available specifications for photographic chemicals of satisfactory quality.

0.3 Benzotriazole is used as a photographic antifogging agent in processing. It is also known as 1, 2, 3-benzotriazole, 1-H-benzotriazole and benzisotriazole. It has the following structural formula:



Molecular Formula $C_6H_5N_3$
(Molecular Mass 119·12)

0.4 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements, and methods of sampling and test for photographic grade benzotriazole used in the processing of sensitized photographic materials.

2. REQUIREMENTS

2.1 Description — The material shall be in the form of white to cream coloured needles or granular powder and shall consist essentially of 1, 2, 3-benzotriazole.

2.2 Appearance of Solution — 1 g of sample shall dissolve in 100 ml of warm water at approximately 50°C to form a clear solution with not more than a pale yellow colour and shall not have considerable amount of suspended and fibrous matter.

2.3 The material shall also comply with the requirements prescribed in Table 1 when tested according to the methods given in Appendix A. Reference to the relevant clauses of Appendix A is given in col 4 of Table 1.

TABLE 1 REQUIREMENTS FOR BENZOTRIAZOLE, PHOTOGRAPHIC GRADE

(*Clauses* 2.3, B-2.1, B-2.2, B-4.1 *and* B-4.2)

SL No.	CHARACTERISTIC	REQUIREMENT	METHOD OF TEST (REFERENCE TO CLAUSE NO. IN APPENDIX A)
(1)	(2)	(3)	(4)
i)	Assay, percent by mass	99 to 101	A-2
ii)	Ash, percent by mass, *Max*	0·3	A-3
iii)	Iron (as Fe), percent by mass, *Max*	0·005	A-4
iv)	Heavy metals (as Pb), percent by mass, *Max*	0·001	A-5
v)	Volatile matter at 70°C, percent by mass, *Max*	0·5	A-6
vi)	Identity	To pass the test	A-7
vii)	*p*H of 1 percent aqueous solution	4·5 to 5·5	A-8

---

*Rules for rounding off numerical values (*revised*).

3. PACKING, MARKING AND SAFETY PRECAUTIONS

3.1 Packing — The material shall be packed in suitable, dark coloured air-tight bottles or as agreed to between the purchaser and the supplier.

3.2 Marking — The container shall be securely closed and legibly and indelibly marked with the following information:

a) Name and grade (photographic) of the material;
b) Mass of the material;
c) Date of manufacture;
d) Manufacturer's name and/or his recognized trade-mark, if any; and
e) Lot number in code or otherwise to enable the batch of manufacture to be traced from records.

3.2.1 The containers may also be marked with the Standard Mark.

NOTE — The use of the Standard Mark is governed by the provisions of the Bureau of Indian Standards Act, 1986 and the Rules and Regulations made thereunder. The Standard Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by BIS and operated by the producer. Standard marked products are also continuously checked by BIS for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the Standard Mark may be granted to manufacturers or producers, may be obtained from the Bureau of Indian Standards.

3.3 Safety Precautions — Benzotriazole is moderately skin irritant.

3.3.1 Normal precautions required while following any chemical procedure must be exercised at all times.

4. SAMPLING

4.1 Representative samples of the material shall be drawn and their conformity to the standard determined as prescribed in Appendix B.

APPENDIX A

(*Clause* 2.3)

METHODS OF TEST FOR BENZOTRIAZOLE, PHOTOGRAPH GRADE

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977*) shall be used in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the result of analysis.

A-2. DETERMINATION OF ASSAY

A-2.0 General — Two methods have been prescribed for determination of assay, namely:

1) Gravimetric method, and
2) Potentiometric titration method.

However, in case of dispute, the gravimetric method shall be the referee method.

A-2.1 Gravimetric Method

A-2.1.0 *General* — Benzotriazole forms a quantitative complex with silver nitrate which is estimated gravimetrically.

A-2.1.1 *Reagents*

A-2.1.1.1 *Ammonia solution* — 10 percent (*m/v*).

A-2.1.1.2 *Silver nitrate solution* — 10 percent (*m/v*).

A-2.1.2 *Procedure* — Weigh accurately 0·25 ± 0·01 g of the sample and dissolve in 10 ml of ammonia solution (**A-2.1.1.1**). Add 50 ml water and heat to a temperature between 60 to 90°C. Add slowly with stirring, 10 ml of silver nitrate solution. Digest at 60°C for 15 minutes, cool to room temperature and filter through a tared sintered glass crucible G-3. Wash the precipitate with six 10 ml portions of water and dry at 105°C to constant mass.

NOTE — Acidify the filterate with nitric acid before disposal.

---

*Specification for water for general laboratory use (*second revision*).

A-2.1.3 *Calculation*

$$\text{Assay (percent by mass)} = \frac{M_2 \times 52{\cdot}70}{M_1}$$

where

M_1 = mass in g of sample, and
M_2 = mass in g of the precipitate.

A-2.2 Potentiometric Method

A-2.2.0 *General* — Benzotriazole solution is titrated with silver nitrate solution and the end point is determined potentiometrically.

A-2.2.1 *Reagents*

A-2.2.1.1 *Sodium acetate solution* — 10 percent (*m/v*).

A-2.2.1.2 *Standard silver nitrate solution* — 0·1 N.

A-2.2.2 *Apparatus* — Potentiometer provided with silver and calomel electrodes with a potassium nitrate bridge.

A-2.2.3 *Procedure* — Weigh accurately 0·5 ± 0·001 g of the sample and dissolve in 100 ml water in a 250-ml beaker. Add 10 ml sodium acetate solution and titrate potentiometrically with 0·1 N silver nitrate. As the end point is reached, add silver nitrate solution in 0·1 ml increments. In a similar manner, titrate a blank consisting of the same reagents as used above but without the sample. Plot the titration curves and determine the inflection points. Subtract the blank titration value from the sample titration value.

A-2.2.4 *Calculation*

$$\text{Assay (percent by mass)} = \frac{V \times N \times 11{\cdot}91}{M}$$

where

V = net volume in ml of silver nitrate solution,
N = normality of silver nitrate solution, and
M = mass in g of the sample taken.

A-3. DETERMINATION OF ASH

A-3.0 General — A known quantity of the material is ignited at 600°C and ash obtained is cooled and weighed.

A-3.1 Apparatus

A-3.1.1 *Silica Crucible*

A-3.1.2 *Furnace* — Electrical furnace with automatic temperature control device.

A-3.2 Procedure — Weigh accurately 5·0 ± 0·1 g of the sample in a tared silica crucible. Moisten it with one or two drops of concentrated sulphuric acid. Incinerate and ignite at dull red heat (about 600°C) to complete the ignition. Cool in a desiccator and weigh to get constant mass.

A-3.2.1 Preserve the ash for determination of iron and heavy metals.

A-3.3 Calculation

$$\text{Ash, percent by mass} = \frac{100 \times M_2}{M_1}$$

where

M_1 = mass in g of the sample taken, and
M_2 = mass in g of the ash.

A-4. DETERMINATION OF IRON

A-4.0 General — Two methods have been prescribed for determination of iron, *o*-phenanthroline method and thioglycollic acid method. However, in case of dispute, the *o*-phenanthroline method shall be the referee method.

A-4.1 *o*-Phenanthroline Method

A-4.1.0 *General* — *o*-phenanthroline, hydroxylamine hydrochloride solution and acetate buffer solution of *p*H 5, in equal quantities are mixed with the material and the colour is compared with that produced by standard iron solution under identical conditions.

A-4.1.1 *Reagents*

A-4.1.1.1 *Acetate buffer pH 5* — Add 23 g of anhydrous sodium acetate to 50 ml of 2 molar acetic acid and dilute to one litre with water. Adjust the final *p*H of the solution to 5 ± 0·1 with acetic acid or 10 percent sodium hydroxide solution.

A-4.1.1.2 *Hydroxylamine hydrochloride* — 10 percent aqueous solution (*m*/*v*).

A-4.1.1.3 *o-phenanthroline (1, 10 — phenanthroline)* — 0·1 percent aqueous solution (*m*/*v*).

A-4.1.1.4 *Standard iron solution* — Dissolve 7·02 g of crystalline ferrous ammonium sulphate [$FeSO_4(NH_4)_2SO_4.6H_2O$] in about 300 ml of water containing 2 ml of concentrated sulphuric acid and dilute to 1 000 ml with water. Further dilute 5 ml to 1 000 ml fresh when required. One millilitre of the diluted solution contains 0·005 mg of iron (as Fe).

A-4.1.2 *Procedure*

A-4.1.2.1 Add 5 ml of concentrated hydrochloric acid to the residue obtained from the determination of ash (*see* **A-3**) and evaporate to dryness. Dissolve the residue in 15 ml of dilute hydrochloric acid and warm on a steam bath for 10 minutes. Cool, dilute to 50 ml with water in a volumetric flask and mix thoroughly. Use 20 ml of this solution for the determination of iron and preserve the remaining solution for heavy metals.

A-4.1.2.2 Thoroughly mix equal parts of *o*-phenanthroline, hydroxylamine hydrochloride and acetate buffer of *p*H 5. Add 5 ml of this mixture to 20 ml portions of both the standard iron solution and the sample solution. Dilute to 50 ml with water and mix well.

A-4.1.2.3 The material shall be taken to have not exceeded the limit prescribed for iron, if the colour produced is not deeper than that produced by the standard iron solution.

A-4.2 Thioglycollic Acid Method

A-4.2.0 *General* — Add concentrated hydrochloric acid to the residue obtained from the determination of ash, evaporate to dryness and finally dissolve in dilute hydrochloric acid. Add thioglycollic acid, make the solution alkaline and compare the colour produced with that produced by standard iron solution under identical conditions.

A-4.2.1 *Apparatus*

A-4.2.1.1 *Nessler cylinders* — 50-ml capacity.

A-4.2.2 *Reagents*

A-4.2.2.1 *Dilute hydrochloric acid* — 10 percent (*v/v*).

A-4.2.2.2 *Thioglycollic acid* — 89 percent purity, *Min.*

A-4.2.2.3 *Dilute ammonium hydroxide* — 1 : 2.

A-4.2.2.4 *Citric acid solution* — 25 percent (*m/v*).

A-4.2.3 *Procedure*

A-4.2.3.1 Transfer a 20 ml aliquot of the sample solution from **A-4.1.2.1** to a Nessler cylinder and dilute to about 30 ml. Into the second Nessler cylinder containing 5 ml of dilute hydrochloric acid, pipette 20 ml of standard iron solution and dilute to about 30 ml with water. To both the cylinders, add 2 ml of citric acid solution and 2 to 3 drops of thioglycollic acid. Mix, make just alkaline with ammonium hydroxide and dilute to 50 ml.

A-4.2.3.2 The material shall be taken to have not exceeded the limit prescribed for iron, if the colour produced is not deeper than that produced by the standard iron solution.

A-5. DETERMINATION OF HEAVY METALS

A-5.0 General — The colour produced by heavy metals in the sample on treating with hydrogen sulphide is compared with that produced with standard lead solution under identical condition.

A-5.1 Reagents

A-5.1.1 *Dilute Ammonium Hydroxide* — 10 percent (*m/m*).

A-5.1.2 *Dilute Hydrochloric Acid* — approximately 0·1 N.

A-5.1.3 *p-Nitrophenol Indicator* — 0·25 percent aqueous solution (*m/v*).

A-5.1.4 *Hydrogen Sulphide* — saturated aqueous solution.

A-5.1.5 *Standard Lead Solution* — Dissolve 0·64 g of the lead nitrate in water; add one millilitre of concentrated nitric acid and dilute to 1 000 ml with water. Further dilute 10 ml to 1 000 ml fresh, when required. One millilitre of the resulting solution contains 0·004 mg of lead.

A-5.2 Procedure

A-5.2.1 To the 20 ml portion of the sample solution (*see* **A-4.1.2.1**) in a Nessler cylinder, add a few drops of *p*-nitrophenol indicator and then add ammonium hydroxide dropwise, until the solution turns yellow. Add dilute hydrochloric acid dropwise, until the solution becomes colourless again and then add 0·5 ml in excess. Add 5 ml of hydrogen sulphide solution and dilute to 50 ml.

A-5.2.2 At the same time transfer 5 ml of standard lead solution into another Nessler cylinder and render it neutral to *p*-nitrophenol indicator by using dilute ammonium hydroxide and dilute hydrochloric acid as described in **A-5.2.1**. Add 0·5 ml of dilute hydrochloric acid in excess and 5 ml of hydrogen sulphide solution. Dilute to 50 ml.

A-5.2.3 The material shall be considered to have passed the requirement of the test if the colour produced in the sample solution is not more intense then that produced in the standard solution.

A-6. DETERMINATION OF VOLATILE MATTER

A-6.0 General — A known quantity of the material is dried at 70°C and loss in mass is taken as volatile matter.

A-6.1 Apparatus

A-6.1.1 *Glass-Stoppered Shallow Weighing Bottle*

A-6.1.2 *Oven* — preferably with automatic temperature control device.

A-6.1.3 *Desiccator* — *see* IS : 6128-1971*.

A-6.2 Procedure — Weigh accurately 3 ± 0·1 g of the material in a weighing bottle and dry in an oven at 70°C for about 8 hours. Cool in the desiccator and weigh.

A-6.3 Calculation

$$\text{Volatile matter, percent by mass} = \frac{(M - M_1)}{M} \times 100$$

where

M = mass in g of the material taken for the test, and

M_1 = mass in g of the material obtained on drying.

A-7. IDENTITY

A-7.0 The material shall pass either melting point test or the infra-red identity test or the ultra-violet identity test.

A-7.1 Melting Point Test — Two methods have been prescribed for melting point test. In case of dispute, the mixed melting point method shall be the referee method.

A-7.1.1 *Individual Melting Point Method*

A-7.1.1.1 *Apparatus*

a) *Capillary glass tubes for melting point* — thin-walled, uniformly bored capillary tubes with one end closed.

b) Capillary tube melting point apparatus with temperature control device.

c) *Thermometer* — 0-110°C (*see* IS : 4825-1982†).

A-7.1.1.2 *Procedure* — Transfer sufficient quantity of fine powder of the sample to the capillary tube to give a column of about 3 mm. Heat the bath of the melting point apparatus to 85°C. Insert the capillary tube to the apparatus so that the closed end is near the middle of the thermometer bulb. Continue to heat the bath at 2°C/min. Note the temperature at which liquefication occurs which is indicated by the formation of a definite meniscus. The melting point should be between 96 to 98°C.

A-7.1.2 *Mixed Melting Point Determination*

A-7.1.2.1 *Apparatus*

a) Capillary glass tube for melting point — thin walled, uniformly bored capillary glass tubes with one end closed.

---

*Specification for desiccators.

†Specification for liquid-in-glass solid-stem reference thermometers (*first revision*).

b) Capillary tube melting point apparatus with temperature control device.

c) Thermometer 0-110°C (*see* 4825-1982*).

A-7.1.2.2 *Procedure* — Prepare three capillary tubes containing:

a) the sample to be tested,

b) a sample known to be benzotriazole, and

c) A finely ground mixture of (a) and (b) mixed in the ratio 1:1.

Identify the capillary tubes. Heat the melting point apparatus to about 85°C and insert the capillary tubes in the apparatus so that the closed end is near the middle of the thermometer bulb and thereafter heat at a constant rate of 2°C/minute. Note the temperature at which liquefication occurs which is indicated by the formation of a definite meniscus. The melting point of the mixture of the sample and standard shall be not lower than the melting point of either the sample or the standard.

A-7.2 Infra-Red Identity Test

A-7.2.1 *Apparatus* — Infra-red spectrophotometer equipped for 2-15 μm region and provision to use pressed potassium bromide pellets.

A-7.2.2 *Procedure* — Grind about 1 g of the sample to a fine homogeneous powder and prepare 0·5 percent (*m/m*) mixture of the sample in potassium bromide. Grind thoroughly to about 200 mesh. Prepare a pressed pellet of the mixture to contain 0·13 to 0·16 g per cm^2 of the material. Record the infra-red spectrum from 2 to 15 μm. Compare with the reference spectrum given in Fig. 1.

A-7.3 Ultraviolet Identity Test

A-7.3.1 *Apparatus* — Ultraviolet spectrophotometer equipped for 200 to 350 μm region.

A-7.3.2 *Procedure* — Prepare 0·002 5 percent solution of the material in water. Record ultraviolet spectrum from 200 to 300 millimicrons against water blank. Compare with the reference spectrum of a sample known to be benzotriazole.

A-8. *p*H OF THE SOLUTION

A-8.0 General — The *p*H of a 1 percent solution of the material is determined by using a suitable *p*H meter.

A-8.1 Apparatus

A-8.1.1 *p*H *Meter* — with glass and calomel electrodes.

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*Specification for liquid-in-glass solid-stem reference thermometers (*first revision*).

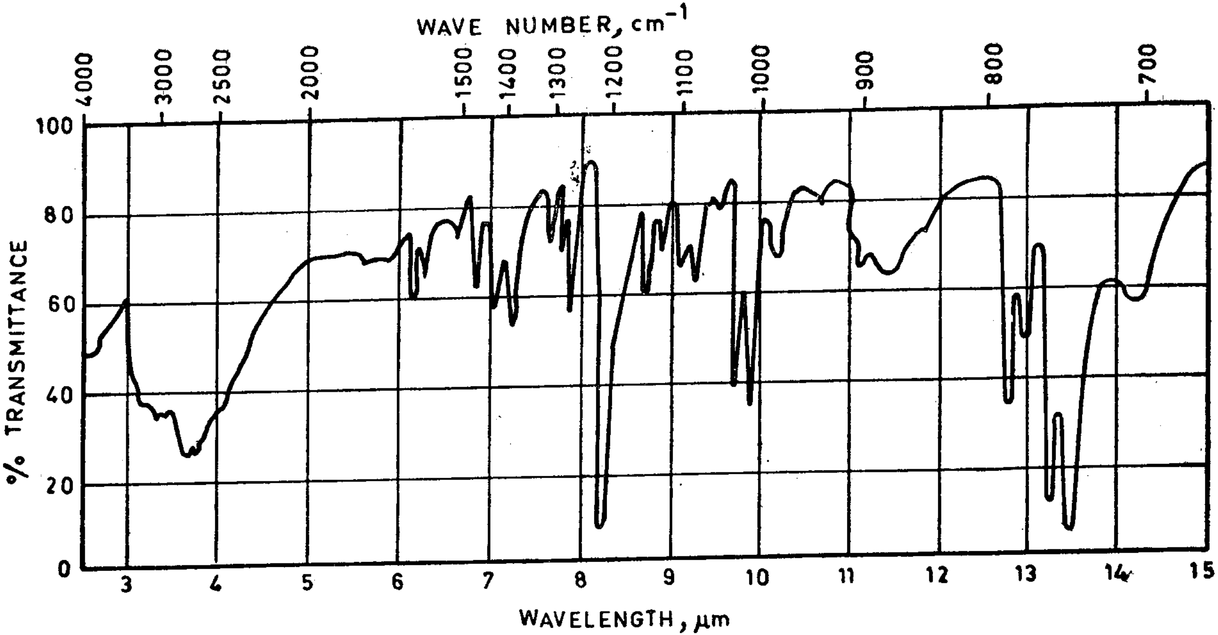

FIG. 1 REFERENCE INFRA-RED SPECTRUM OF BENZOTRIAZOLE (KBr PELLET)

A-8.1.2 *Procedure* — Prepare a one percent solution of the material in a 100-ml beaker, using freshly boiled and cooled water. Measure the *p*H of the solution using a *p*H meter.

APPENDIX B

(*Clause* 4.1)

SAMPLING OF BENZOTRIAZOLE, PHOTOGRAPHIC GRADE

B-1. SCALE OF SAMPLING

B-1.1 Lot — In any consignment, all the packs manufactured in a single batch shall constitute a lot.

B-1.1.1 In order to ascertain the conformity of the lot to the requirements of this specification, samples shall be tested for each lot separately.

B-1.2 The number of packs to be chosen from the lot shall depend upon the size of the lot and shall be in accordance with Table 2.

TABLE 2 NUMBER OF PACKS TO BE SELECTED FOR SAMPLING

LOT SIZE (1)	SAMPLE SIZE (2)
Up to 50	3
51 to 100	4
101 to 150	5
151 and above	7

B-1.3 These packs shall be chosen at random from the lot and in order to ensure the randomness of selection, the procedure given in IS : 4905-1968* may be followed.

B-2. NUMBER OF TESTS

B-2.1 Tests for the requirements of assay content, percent by mass, given in Table 1 shall be conducted on individual sample.

B-2.2 For all other requirements given in Table 1 and **2.2**, tests shall be conducted on composite sample.

---

*Methods for random sampling.

B-3. PREPARATION OF COMPOSITE SAMPLES

B-3.1 Benzotriazole shall be taken from each selected pack in required quantities in a container and shall be mixed thoroughly for preparing a composite sample.

B-4. CRITERIA FOR CONFORMITY

B-4.1 The lot shall be considered satisfactory with respect to assay content, if the following conditions are satisfied:

From the test results, the average ($\bar{X}$) and range (R) shall be calculated as:

$$\text{Average } (\bar{X}) = \frac{\text{Sum of test results}}{\text{Number of tests}}$$

Range (R) = difference between the maximum and minimum values of test results

The value of expression $\frac{R}{U - L}$ shall be $\leqslant 0{\cdot}9$

where

$\bar{X} + 0{\cdot}6R \leqslant U$, and
$\bar{X} - 0{\cdot}6R \geqslant L$.

U and L are the upper and lower specifications limits given in Table 1.

B-4.2 For the remaining characteristics tested on composite sample, the results shall meet the relevant requirements given in Table 1 and **2.2**.

INTERNATIONAL SYSTEM OF UNITS (SI UNITS)

Base Units

QUANTITY	UNIT	SYMBOL
Length	metre	m
Mass	kilogram	kg
Time	second	s
Electric current	ampere	A
Thermodynamic temperature	kelvin	K
Luminous intensity	candela	cd
Amount of substance	mole	mol

Supplementary Units

QUANTITY	UNIT	SYMBOL
Plane angle	radian	rad
Solid angle	steradian	sr

Derived Units

QUANTITY	UNIT	SYMBOL	DEFINITION
Force	newton	N	$1 \ N = 1 \ kg.m/s^2$
Energy	joule	J	$1 \ J = 1 \ N.m$
Power	watt	W	$1 \ W = 1 \ J/s$
Flux	weber	Wb	$1 \ Wb = 1 \ V.s$
Flux density	tesla	T	$1 \ T = 1 \ Wb/m^2$
Frequency	hertz	Hz	$1 \ Hz = 1 \ c/s \ (s^{-1})$
Electric conductance	siemens	S	$1 \ S = 1 \ A/V$
Electromotive force	volt	V	$1 \ V = 1 \ W/A$
Pressure, stress	pascal	Pa	$1 \ Pa = 1 \ N/m^2$